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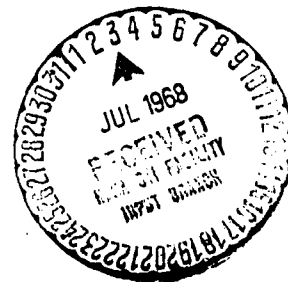
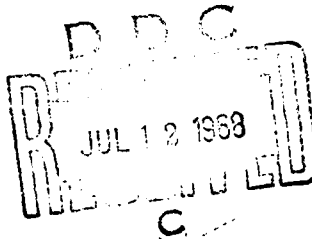
## TECHNICAL REPORT

ONRL-41-68

MATERIALS RESEARCH IN ISRAEL

By HARRY A. LIPSITT

14 June 1968



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## MATERIALS RESEARCH IN ISRAEL

### INTRODUCTION

The materials sciences scene in Israel has changed only marginally since J.B. Cohen's visit of December 1966 (see ONRL-5-67). Some other factors have changed markedly. The ten-mile-wide corridor between Haifa and Tel Aviv is now fifty miles wide. Inflation continues. The military aspects of the country are everywhere visible -- only now the main means of military movement is via Russian trucks.

I find myself in complete agreement with Cohen's comment that the magnificent buildings are only adequately equipped. On the other hand, everyone seems to be doing Mössbauer spectroscopy using the Israeli developed and manufactured ELRON electronics package -- they have, perhaps, "compensated" for the lack of general facilities by moving en masse in the Mössbauer direction. I cannot reinforce strongly enough Cohen's views on the relative needs of the country regarding university vs. industrial expansion. But I might go even a step further; at this time, when outside support for research is tenuous, at best, it is clear that they are too heavily committed to non-remunerative, non-directional, non-time oriented research.

On this trip I was able to visit the Technion, the Weizmann Institute and the Israel Atomic Energy Commission. I spoke to some of the same people that Cohen did 18 months before and in those cases only a progress report will be given.

### Technion - Israel Institute of Technology, Haifa

I first spoke briefly to Prof. J. Hagin (pronounced Hageen), Director, Technion Research and Development Foundation Ltd. Several interesting facts came out of this briefing: First, they hope to rename the Technion soon and call it the Technological Institute of Israel. This they will do as soon as there are other engineering schools in the country. The second thing that I was interested in was the fact that they professed to educate engineers and scientists to the level of the advanced countries. They do this because "this little country," in their words, does not have very much in the way of resources, does not have cheap power, and therefore their only resource is educated brains, of which they need to make considerable use.

The Technion staff has grown somewhat since Cohen was here; they now have about 800 on the staff. They encourage consulting work, but I have learned that the consulting is done mainly by the people with more applied interests and few of those more fundamentally inclined are involved.

The graduate student body is now of the order of 1500, of which about one-sixth are working towards doctorates and the rest toward masters. Interestingly enough, about 80% of those working for master's degrees are not full-time students, but are people employed all over Israel who put in one day a week, or perhaps more, at the Technion doing their master's degree over a considerably longer period of time -- usually about four years. When I asked in the departments about this scheme I learned that the industrial people are pretty harassed at work and they cannot always spend more than the minimum amount of time doing their theses. They do attend the classes and benefit this way, but their thesis work is sometimes a bit haphazard and sometimes not very much is done at all. Since the Technion specifies that the master's thesis should be an original contribution, this has led to a double standard, with only the full-time students living up to the standard. They use a technique we have seen elsewhere: a piece of equipment that they need is built, calibrated and checked out by a student who does a little bit of work on it and who then receives his master's degree for that; another man comes along and uses that piece of equipment for a good piece of research for which he receives a PhD.

Another interesting facet is that the by-laws of the Technion actually state that every faculty member must spend one-quarter to one-third of his time doing research. I checked this with David Brandon, because he is an exceptional man with an abundance of energy, and if any man were able to stick to this by-law, I figured he could. Brandon's answer was no, he cannot do any research himself, he has no time, what with teaching duties, graduate students, preparing and giving lectures, all the committees and the fact that he does spend one day a week at the atomic energy establishment at Beersheba. So things are not very much different here than anywhere else.

#### The Department of Materials Engineering and the Israel Institute of Metals

Prof. David Brandon (Cambridge PhD with Nutting, then field ion microscopy at Cambridge and Battelle, Geneva, before moving over to the Technion about 18 months ago) told me about the teaching activities of the Department. What has happened effective 1 April 1968 is that the Department of Metallurgy and the Department of Solid State Mechanics have been combined to form a new department called Materials Engineering, which is still within the Faculty of Mechanical Engineering. Prof. Sol Bodner, who was the head of Solid State Mechanics, is the head of this new Department. Although the new Department is formed it is not yet put together in one place, and they see very little of Bodner. However, the curriculum changes that they wanted have been accomplished, and so the metallurgy teaching duties now consist of a compulsory materials course given to all second year engineering students, and a compulsory metallurgy course which is given to third year mechanical engineering students. The newest innovation is that there is now a materials engineering option available to fourth year mechanical engineering students. They expect about one-fifth of the total of 150 mechanical engineering students to elect this option. This is the first true metallurgy or materials curriculum is being offered in Israel. On the graduate level they handle about ten graduate students per year, mainly for the two year master's degree. For this they offer a full range of courses.

Brandon's research interests are concentrated in three lines. He is interested in the development of electrographic recording for use in field ion microscopy (FIM). The reason for this is that for his work in FIM, he generally has far more resolution in the beam and even on the phosphor screen than he is able to get in the film used to photograph the screen. So, he has been looking for a much better way, a way with higher resolution, for use in FIM. Films are hopeless because they continually outgas when they are in the FIM vacuum system. They have been working mainly on electrophoretically deposited colloidal zinc oxide plates which, Brandon says, have a resolution of about 1200 lines per mm! They have been attempting to measure the response of plates made either of anodized aluminum or zinc oxide. To measure the response of these plates, images are formed using various imaging gases in the microscope. He is also going to try tantalum oxide as a recorder film because this has a higher dielectric constant and should give even better results than does zinc oxide.

His second project is the influence of combined stresses on dislocation substructure, and this has really led to two programs. One is to study the influence of heat pulses on the recovery of a plastically deformed material. They have found, so far, that they get a much more uniform distribution of dislocations in the case of the use of heat pulses than if a constant temperature were used. Brandon is not sure of the reason for this, but he thinks that it might be due to the fact that one gets a supersaturation of vacancies each time a heat pulse is applied and these will influence the kinetics of the recovery of the cold worked material. As an example of how this works they have found that in tantalum one obtains fairly good recovery at a steady temperature of 1200°C, whereas the same structure can be obtained using a series of pulses of which the maximum temperature of any pulse is 800°C.

He also has a man doing a PhD thesis studying the influence of the combination of creep and fatigue at high temperatures -- temperatures to 1200°C. Here they are really looking for the influence of pulsating stress on a structure that is undergoing a constant rate of extension as it does in creep.

The third project, in which Brandon is very much interested, is the crystallization of oxide films which are amorphous as deposited. They are looking at electrolytically formed metastable oxides as they are heated and begin to crystallize. He has been studying the oxides of Al, Ta, W, Nb and Ti and he finds the following sorts of things. The oxide  $\text{Nb}_2\text{O}_5$  crystallizes as an orthorhombic, but a much simpler orthorhombic cell than that reported in the literature.  $\text{TiO}_2$  is the only one they have studied which is deposited as a crystalline phase and it is in the tetragonal form called anatase. The aluminum oxide  $\text{Al}_2\text{O}_3$  crystallizes as the cubic gamma alumina. The tungsten case is very interesting in that the as-deposited film consists of two layers. The layer next to the metal has the composition  $\text{WO}_2$  and crystallizes as the monoclinic. The layer on the outside is  $\text{WO}_3$  but it is actually the beta  $\text{WO}_{2.9}$  and is a pseudo primitive cubic cell.

When the oxides are crystallized by heating they find that nucleation is the rate controlling step in the  $Ta_2O_5$ ; they get a sharp boundary between the crystallized material and the amorphous material. In the  $Al_2O_3$  nucleation is easy, but the growth step is difficult; so many grains are nucleated, but they do not grow very much and one ends up with a lot of small grains of crystalline oxide. With the  $Nb_2O_5$  there is no sharp boundary existing between the crystallized and the amorphous portions of the material and a diffraction pattern of this structure shows considerable streaking. Brandon's description of the way in which this crystalline material grows is rather interesting. He describes it as being a reorganization of the atoms rather than recrystallization. The reorganization occurs first in one direction in the lattice and then in another direction, etc., until the material is fully crystallized. The situation in the tungsten oxides is also interesting because of the fact that there are the two oxides formed. When the crystallization occurs one gets a eutectoid structure with the  $WO_2$  forming the dendrites and the  $WO_3$  filling in the interdendritic spaces.

David Brandon and Abraham Rosen, to whom I talked next, are putting on a symposium as one of the means of getting people here to see Israel, etc., but also because David, having now been here a little more than a year, is painfully feeling the lack of communication with his colleagues in Europe and America. This is one way of getting to talk to them, because he cannot get out to see them as often as he would like. The conference is on the Quantitative Relations Between Properties and Microstructure and will be held in Haifa on 27 July through 1 August 1969.

Abraham Rosen got his PhD in the Technion and then, as he said, went to work for John Dorn to learn something. This is a little different story than Prof. Hagin told about their PhD. Rosen has been working on repeated yielding of aluminum and other materials. He has worked on commercial purity aluminum, high purity aluminum, stainless steel, brass, duralumin, etc. Although he has worked in fairly close contact with Brandon for more than a year, he apparently has not yet been convinced to do any electron microscopy, and he has also been very heavily influenced by Prof. Bodner, whose field is mechanics of continuous media. So, the explanations that he has for discontinuous yielding do not involve the structure of the material at all. Anyway, he predicts serrated yielding under any conditions where the stress versus strain rate curve shows decreasing stress with increasing strain rate for a given strain, but he does not know why.

Rosen has a student working on superplasticity. He is taking a new experimental approach to the problem in that he is not even attempting to keep the whole specimen extending in the region of uniform strain and uniform extension. Rather he has built what is essentially a one turn furnace and will study superplasticity over a very small gauge length and look at the microstructures, etc. He is thinking of starting by studying Fe near the  $\alpha$ - $\gamma$  phase transformation.

I finished the day by talking to Dr. S. Niedzwiedz, from the metallurgy department, and Dr. M. Ron, from the physics department, who have been doing

combined research using the Mössbauer Effect to clarify the nature of the carbides which appear in steel as it is being tempered. The carbides which have been variously supported (and not supported) in the literature are the c.p.h. Eta carbide which shows a Curie temperature of 380°C, the Chi (or monoclinic) carbide which shows a Curie temperature of 285°C, and the Theta orthorhombic carbide (cementite) which has a Curie temperature of 210°C. These gentlemen examined filings from steel specimens which had been quenched to martensite and tempered at various temperatures for various times. They used Mössbauer spectroscopy particularly to find the doublet which is characteristic of paramagnetic or superparamagnetic materials and watched this doublet disappear with increasing tempering temperature (the doublet, of course, being characteristic of the eta carbide). They found that as the doublet disappears, the ferromagnetic lines of cementite, the orthorhombic theta carbide, appear smoothly to take its place showing the six peaks characteristic of a ferromagnetic material. This proves, they professed, that the chi monoclinic carbide does not exist at all and that instead there is a smooth transition between the eta and the theta.

The next day I visited the Israel Institute of Metals, which is the arm of the Technion that does research and development, testing, and standardization that is needed by Israeli industry. I spoke first to the director, Mr. S. Goldschmidt. He told me there were five divisions within the Institute, having to do with performance, standards, testing, etc. One of these is the corrosion and surface treatment section, under the leadership of Dr. J. Yahalom. Yahalom did his PhD in Cambridge and has been in the Technion ever since. The foundry section does research on the influence of molding sand on the surface quality of castings, makes formulations for the sand mixtures to be used in castings, formulations using sands indigenous to various parts of Israel. The other sections are the general metallurgy and metallography section, nondestructive testing and environmental tests, etc.

Yahalom is working with his PhD students studying the reversal of polarity of cadmium plated steel in some situations; when this happens, the cadmium plate becomes cathodic and is no longer corrosion protection for the steel underneath. This happens sometimes in certain carbonate solutions and they are investigating the mechanism of this reversal.

Yahalom is a member of both the Institute of Metals and of the Metallurgy Department, so part of his work is practical. A more practical program that they are working on is a study to determine if stainless steel cutlery can be finished by electropolishing efficiently and competitively. They find that it can be done, but it is more expensive than mechanical finishing because of the cost of regenerating the polishing solution as time goes on. They are trying to introduce an ion exchange method to constantly regenerate the solution and thus make the electropolishing technique cheaper than mechanical polishing. I asked Yahalom what the finish was like and, as you would expect, it is shinier than the mechanical finish, but he says it is perfectly acceptable and the choice is then merely a matter of taste.

Yahalom is also developing an instrument to follow the process of sealing, one of the steps in the anodizing of aluminum. Sealing is a step in which the anodized aluminum is put into some water base solution and the water diffuses into the aluminum oxide and hydrates it, causing it to expand, which seals the pores in the oxide. To follow the course of the sealing one uses a small voltage at about 1 kc frequency across the test piece, and using a resistance-capacitance bridge, the impedance change during the sealing operation is measured until a previously determined, acceptable impedance value is obtained. When the predetermined impedance is achieved, the anodized coating has been sealed quite properly and will pass any test that one might wish to give. This method of monitoring the sealing process while it occurs is much faster than the salt spray test (which would take a week or so) or the ink absorption test which can also be used to see whether any more liquid can be absorbed. And finally, this technique would seem to be a little bit better than the use of the Zetascope (sold by Twin Cities Instrument Company in the United States) for the simple reason that this device is used during the sealing operation, whereas the Zetascope is only useful after the sealing.

Goldschmidt is not only the Director of the Israel Institute of Metals, but is also the head of the Nondestructive Testing Group. That Group has been attempting to develop thermographic techniques for finding unbonded areas in composite structures. They use a resistance bridge of which one branch contains a thermister. Thus, if there is a very slight change in the heat transfer across one area of the specimen (due to a poor bond) one sees a great change in the resistance of the thermister which is very easily picked up. They are also developing a technique to test the bonding of honeycomb structures by back scattering of radiation.

After leaving Goldschmidt I went to talk to Prof. Sol Bodner. Bodner and Abraham Rosen have been studying serrated yielding. Bodner showed me the machine that they are using, which albeit primitive, was very good for what they wanted here. The specimen was loaded slowly, but uniformly, by the flow of water into a drum at constant rate to give a constant loading rate. A standard testing machine yields constant cross-head speed. The difference between these results is fairly obvious. Where serrated yielding is studied with a standard testing machine one observes a load drop when the specimen suddenly yields. On the other hand, in a machine operating at constant loading rate, when the specimen suddenly yields, the load remains constant and the yield strain is seen. So, whereas the first mentioned stress-strain curve has wiggles on it that go up and down, the second kind of curve actually is a step function composed of sudden strains connected by plastic stress increases.

Bodner is simultaneously measuring the strain rate and the velocity of the stress wave that passes down the specimen using bonded wire gauges, and attempting to relate these to dislocation velocity. With this information he can solve the Gillman-Johnston equation to get the density of mobile dislocations. He has used this information in a theory which he has developed and published and which he is now testing (Mater. Sci. Eng., 2, 213 (1967)).

An example of other work going on in Bodner's laboratory is the influence of impacting on spalling of composites. To study this they fire a rifle bullet into a composite and use a ballistic pendulum to determine the transfer of momentum and then, of course, they examine the composite to see how it has been spalled. They are also studying the creep of glass fiber reinforced plastic composites to about 100-200°C. He has also got a home-made damping setup and a stress relaxation device in which the extension is fixed and the decay of the load is monitored.

#### The Weizmann Institute, Rehovoth

I was able to visit only the Electronics Department of the Weizmann Institute, headed by Prof. E.H. Frei. Frei has continued his research into magnetic applications. He has been studying the use of barium ferrite as a tracer and indicator in medical work. He points out that this is sometimes considerably better than the barium sulfate which is ordinarily used for gastro-intestinal studies for the reason that the barium sulphate moves continuously through the GI tract and if the physician finds something he wants to study further, it is too late because the barium sulphate has already moved on. On the other hand, using barium ferrite (which is magnetic) as a tracer, the physician may let this pass on down the GI tract until he finds something he wants to study. He can hold the ferrite there with a magnet while he further studies that area. Finally, if some part of the intestine is doubled over, shielding another part from view, a magnet will move one fold of the intestine out from under the other fold, separating the two items to be observed. Frei showed us some pictures of his own innards where this has been used. He was the first volunteer because of the potential danger from unreacted barium.

I spoke next to Dr. S. Shtrikman who pointed out that in the physics or electronics segment of the Weizmann Institute there were about 15 people working on magnetism. The group is guided mainly by Shtrikman and Dr. David Trevis, who has just returned from working in the United States for a couple of years. Shtrikman has been studying magnetic ionic crystals using Mössbauer techniques, ESR, X-rays, optics and specific heat measurements. He has recently published a paper (J. Appl. Phys. 38, 3981 (1967)) in which changes in the Mössbauer spectrum were interpreted in terms of the phase diagram of an Fe-Co-V alloy to lend support to the hypothesis that the magnetic properties are due to the fact that the structure consists of ordered small magnetic domains separated by a paramagnetic phase (as in Alnico type magnets).

His more recent Mössbauer work (submitted to Solid State Communications) is even more interesting. He had been studying the isomer shifts of  $\text{Eu}^{+2}$  and  $\text{Eu}^{+3}$  in bcc  $\text{Eu}_3\text{S}_4$ . One expects to find two well-resolved resonance lines in the  $\text{Eu}_3\text{S}_4$  spectra. The expected ratio of the peak intensities should be equal to the number of  $\text{Eu}^{+2}$  and  $\text{Eu}^{+3}$ , namely 1:2. This is what one finds in the Mössbauer spectra of  $\text{Eu}_3\text{O}_4$ . But  $\text{Eu}_3\text{S}_4$  shows two well-resolved peaks only below 210°K. At higher temperatures the two lines widen and then merge (with increasing

temperature) to form one narrow resonance peak at the center of gravity of the two low temperature peaks. Shtrikman uses an electron hopping model to explain these results. He considers the extra electron of the  $\text{Eu}^{2+}$  ions as localized in a given lattice site, instead of using the concept of energy bands. The electrons move through the lattice via the  $\text{Eu}^{3+}$  ions, overcoming an energy barrier as they do so and remaining bound to each ion for a finite time,  $\tau$ , between jumps. Increasing the temperature enhances the jump probability and lowers the relaxation time,  $\tau$ . Thus, a temperature can be obtained at which the electrons are moving rapidly enough so that the original peaks do not appear and all that shows is a peak which defines the totality of the conductivity electrons. Electrical resistivity checks confirm that both processes have the same activation energy, 0.24 eV. From this, and from the fact that no residual Mössbauer peaks remain at elevated temperatures, Shtrikman concludes that all the "extra" electrons of the  $\text{Eu}^{2+}$  ions take part in the jumping process and that the charge carrier concentration is constant, i.e., the variation in electrical conductivity is due to variations in electron mobility.

#### Soreq Nuclear Research Center, Yavne

This organization has grown considerably since it was visited in 1963 by Lieberman. The swimming pool reactor is now fully active at 5 megawatts and they are planning to double this in the near future. The other buildings are modern and well equipped. The reactor itself is housed in an award-winning building and the whole effect would be quite pleasant if the Center were not stuck away in the desert between Yavne and Ashdod. One word of warning to any visitors: the pile of bricks across from the reactor, that looks like a chunk of Roman ruins, is a donated modern sculpture. Sculpture may have come full circle: Lipsitt still has the dreaded foot-in-mouth disease.

Prof. S. Yiftah is the Director of Soreq but spends his time as the leader of the Reactor Physics Department. Prof. G. Ben-David is head of the Physics Division and Prof. S. Amiel leads the Chemistry Division. Physics consists of Nuclear, Solid State, and Reactor Physics Groups. After an orientation briefing by Mr. M. Morahg, the Administrative Director of Soreq, I spent the balance of my visit with Dr. David Zamir, Acting Head of the Solid State Physics Department.

Zamir spent some time at Cornell University where he studied diffusion in transition metal hydrides. At Soreq he has used nuclear magnetic resonance (NMR) to study the Ti-H system under the sponsorship of the Air Force Materials Laboratory. He measures the nuclear relaxation time which is related to the diffusion coefficient,  $D$ . When there is a jump in the curve of  $D$  as a function of temperature, it means that you have traversed a phase boundary. In this way he has mapped the phase diagram and determined the diffusion coefficient  $D$  and  $\Delta H$ , the activation energy for diffusion for both the alpha and the gamma phases in the Ti-H system. These results were presented at the International Conference on the Strength of Metals and Alloys, Tokyo, September, 1967.

As part of his current work Zamir hopes to do an experiment that will perhaps provide proof that hydrogen agglomerates in some way under the action of the stress and, hopefully, the way in which it will agglomerate may validate one or the other theories of slow strain rate hydrogen embrittlement. The experiment is essentially to do NMR studies on a sample of titanium (probably a bundle of wires) containing hydrogen randomly distributed through the titanium. When there is a random distribution of solute, NMR yields a sharp line. Under the action of the stress this sharp line will become diffused if the hydrogen has agglomerated. There is another very interesting way to do this experiment. The usual specimen for NMR techniques is a powder specimen because this gives the best results. If one uses a thick specimen, you essentially look at just the surface of the specimen. One can then do this experiment with a larger Ti-H specimen, first under no stress and then under the action of a torsion stress. This generates the maximum stress at the surface of the specimen where the nuclear magnetic resonance technique is useful. Zamir is also interested in a study of the way in which alloying affects hydrogen embrittlement in titanium. It is known that the embrittling effect of hydrogen is much less serious in an alpha stabilized material such as Ti-Al. Zamir believes there are two possibilities in this case. One is that when the aluminum stabilizes the alpha it may tremendously increase the solubility of hydrogen in the alpha and avoid the agglomeration in this way, or it is also possible that the diffusion coefficient may increase rapidly due to the addition of the Al giving the appearance in the NMR spectra of a random solid solution. Zamir is also using NMR to study the structure and the defects present in solid and liquid copper up to temperatures of 1200°C.

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<p>Visits to three Israeli institutions are reported. One of these, the Technion, is the only engineering school in Israel. The other institutions visited were the Weizmann Institute and the Soreq Nuclear Research Center of the Israel Atomic Energy Commission.</p>		

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**14. KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, roles, and weights is optional.

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